PATENT ABSTRACTS OF JAPAN

(11)Publication number : 04-006173

(43)Date of publication of application : 10.01.1992

(51)Int.Cl. C04B 37/00
B23K 35/14
B32B 15/01
C22C 9/06
C22C 19/03

(21)Application number : 02-105802

(22)Date of filing : 20.04.1990

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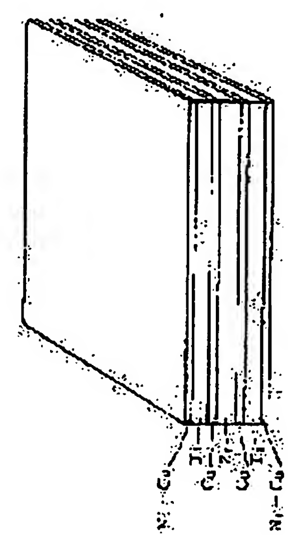
(54) COMPOSITE FOIL BRAZING MATERIAL FOR JOINING CERAMICS

(57)Abstract:

PURPOSE: To provide the above composite foil brazing material which has excellent brazing workability and with which high strength of adhesion is obtainable by coating both surfaces of the Zr material which a core material with 3 laminated materials consisting of a specific compsn. in such a manner that an Ni-Cu material is the outermost surface and the sectional area ratios of the respective blank substrates attain specific values.



CONSTITUTION: Both surfaces of the Zr material which is the core material are coated and formed with the 3 laminated materials consisting of 20 to 70R Cu/Ti/Ni-Cu as a skin material in such a manner that the outermost surface is the 20 to70wt% Ni-Cu material. Further, the materials are so formed that the sectional area ratios thereof attain 30 to 40% Zr material, 35 to 45% Ti material and 10 to 20wt.% Cu material, 20 to 70wt.% Ni-material, 5 to 15% Cu material, by which the composite foil brazing material for joining ceramics consisting of the 7-layered composite material is formed. The brazing material is as relatively low as 900°C in brazing working temp. and, therefore, the brazing

work can be carried out with good workability.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

⑲日本国特許庁（JP）　⑪特許出願公開

⑫公開特許公報（A）　平4-6173

⑮Int.Cl.⁵	識別記号	庁内整理番号
C 04 B 37/00	B	7202-4G
B 23 K 35/14	A	8719-4E
B 32 B 15/01	H	7148-4F
C 22 C 9/06		8015-4K
19/03	G	8928-4K

㊸公開　平成4年(1992)1月10日

審査請求　未請求　請求項の数　1　（全4頁）

㊹発明の名称　セラミックス接合用複合箔ろう材

㉑特　願　平2-105802

㉒出　願　平2(1990)4月20日

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明　細　書

1.発明の名称

セラミックス接合用複合箔ろう材

2.特許請求の範囲

1

芯材のZr材の両面に、最外面がNi20～70wt%-Cu材になる如く、Cu/Ti/Ni20～70wt%-Cuからなる3積層材を外皮材として被覆して形成した7層複合材からなり、前記Zr材、Ti材、Cu材及びNi20～70wt%-Cu材の断面積比率が、

Zr材　30%～40%

Ti材　35%～45%

Cu材　10%～20%

Ni20～70wt%-Cu材　5%～15%

を有することを特徴とするセラミックス接合用複合箔ろう材。

3.発明の詳細な説明

産業上の利用分野

この発明は、セラミックスを接合する箔状の複合ろう材に係り、Zr材を芯材としてその両面に、Cu/Ti/Ni20～70wt%-Cuの外皮材を被覆した7層複合箔ろう材に構成することにより、ろう付け作業温度が900℃と比較的低温であり、ろう付け作業性にすぐれ、高い接着強度が得られるセラミックス接合用複合箔ろう材に関する。

従来の技術

一般に、機構部品、電気·電子部品等においては、セラミックス同志の接合には、その対向面に熱膨張係数が近似し、且つその濡れ性の良好なMo、W等の金属をメタライジングした後、前記メタライジング面上にAgろう付けして接合していたが、メタライジング、及びAgろう付けに多大の手間、コストを要し、特に、接合表面に凹凸を有する異形のセラミックス板への接合には多くの問題点があった。

この発明は、かかる現状に鑑み、セラミックス同志の接合に際し、ろう付け作業温度が低く、かつ取扱いが容易で接着作業性にすぐれ、高い接着強度が得られるとともに、接着コストを低減できるろう材の提供を目的としている。

発明の概要

発明者らは、機構部品、電気·電子部品等におけるセラミックス同志の接合コストの低減を計り、ろう付け作業温度が低く、かつろう付け性にすぐれたろう材を目的に、取扱いが容易と考えられる箔状のろう材について種々検討した結果、Ti材、Zr材、Cu材及びNi20~70wt%-Cu材を用い、これら材料の組合せを選定し、圧接にて特定の断面積比率となした複合箔材が、機構部品、電気·電子部品等におけるセラミックス同志のろう材として、比較的低温ですぐれたろう付け性を発揮し、かつ安価に提供できることを知見し、この発明を完成したものである。

すなわち、この発明は、

芯材のZr材の両面に、最外面がNi20~70wt%-Cu材になる如く、Cu/Ti/Ni20~70wt%-Cuからなる3積層材を外皮材として被覆して形成した7層複合材からなり、前記Zr材、Ti材、Cu材及びNi20~70wt%-Cu材の断面積比率が、

Zr材　30%~40%

Ti材　35%~45%

Cu材　10%~20%

Ni20~70wt%-Cu材　5%~15%

を有することを特徴とするセラミックス接合用複合箔ろう材である。

この発明による複合箔ろう材は、セラミックスとの濡れ性が良好で、セラミックス同志のろう付けに最適であり、従来のAgろう材の如き事前にセラミックスへのメタライジング工程が不要となり、さらに、ろう付け作業温度が900℃と比較的低温であり、作業性にすぐれ、高い接着強度が得られる箔状複合ろう材である。

また、この発明による複合箔ろう材は、同材質同志の接合、あるいは異材質同志の接合でも、いずれの酸化物系セラミックス同志の接合が可能である。

発明の構成

この発明によるセラミックス接合用複合箔ろう材は、Zr材を芯材としてその両面に、Cu/Ti/Ni20~70wt%-Cu の外皮材を被覆した

Ni20~70wt%-Cu/Ti/Cu/Zr/Cu/Ti/Ni20~70wt%-Cuの7層複合箔ろう材であり、第1図に示す如く、最上面層を第1層、最下面層を第7層とすると、下記第1表の如く積層されている。

第1表

3層材(第1,2,3層)	芯材(第4層)	3層材(第5,6,7層)
Ni-Cu－Ti－Cu	Zr	Cu－Ti－Ni-Cu

この発明において、複合箔ろう材として第4層の芯材にZrを用いる理由は、外皮材の3積層材中のTi材(第2層、第6層)と共に活性金属として、セラミックスとの接合に寄与するだけでなく、Ti-Cuの融点を更に低下させるために必要である。

Ti材(第2層、第6層)は、上記の如く、活性金属としてセラミックスと反応して、所要の接着強度を得るために必要である。

また、この発明において、第4層の芯材のZr材との接触面(第3層、第5層)にCu材を用いる理由は、Tiの融点を低下させるために必要なためである。

また、この発明において、外皮材の最外面(第1層、第7層)にNi20~70wt%-Cu材を用いる理由は、Tiの融点を低下させるために必要であると同時に、セラミックスの接合強度を高めるために必要であり、Ni-Cu材のNiが20wt%未満では、接合強度の向上効果がなく、Niが70wt%を越えると、Tiの融点が低下せず作業温度が高くなり好ましくない。

さらに、この発明の複合箔ろう材を構成しているZr材(第4層)、Ti材(第2,6層)、Cu材(第3,5層)及びNi20~70wt%-Cu材(第1,7層)の断面積比率を限定した理由は、Zrが30%未満、Tiが35%未満、Cuが20%を超え、Ni20~70wt%-Cuが15%を超えると、セラミックスとの反応が起こり難く、接合できない問題があり、また、Zrが40%を超え、またTiが45%を超え、Cuが10%未満、Ni20~70wt%-Cuが5%未満では融点が高くなり、作業性が悪

く、セラミックスに対する濡れ性が悪くなるので、好ましくない。

この発明による複合箔ろう材は、例えば、以下の製造方法にて得られる。

コイル状Cu板及びコイル状Ni20~70wt%-Cu板を巻戻し、また、コイル状Ti板を巻戻しながら、前記Ti板を中間層にして、前記Cu板及びNi20~70wt%-Cu板を上方及び下方より板状に圧接しながら、Cu/Ti/Ni20~70wt%-Cuの3層クラッド板を作製する。その後、焼鈍を行なう。

さらに、得られた前記のコイル状Cu/Ti/Ni20~70wt%-Cuクラッド板を巻戻し、また、コイル状Zr板を巻戻しながら、前記Zr板を中間層にして、Cu/Ti/Ni20~70wt%-Cuクラッド板を上方及び下方より、最外面がNi20~70wt%-Cu材になる如く、板状に圧接しながら7層クラッド板を作製する。

その後、焼鈍、圧延を繰返し、所定寸法及び所定断面積比率の複合箔ろう材に仕上げる。

この発明の複合箔ろう材としては、厚みは30~100μmの箔が適当であり、被接着材の種々の形状に容易に馴染み、すぐれた接着性が得られるほか、接着作業が容易になる等の利点がある。

また、この発明の複合箔ろう材を、Ag材で被覆することにより、セラミックスへの濡れ性を著しく改善することができる。

実施例

板厚0.4mm、板幅250mmのTi板、板厚0.18mm、板幅250mmのCu板、及び板厚0.08mm、板幅250mmのNi31wt%-Cu板を巻戻しながら、Ti板を芯にして両面にCu板及びNi31wt%-Cu板を当接させて、圧延率65%で圧接し、板厚0.23mm、板幅250mmのCu/Ti/Ni31wt%-Cuの3層クラッド板を得た。その後、圧接界面の接合強度を高めるため、拡散焼鈍を施した。

次に、板厚0.24mm、板幅250mmのZr板、及び前記の板厚0.23mm、板幅250mmのCu/Ti/Ni31wt%-Cuの3層クラッド板を巻戻ししながら、Zr板を芯にして両面に3層クラッド板を当接させて、最外面がNi31wt%-Cu材になる如く、圧延率65%で圧接し、板厚0.25mm、板幅250mmの7層クラッド板を得た。

その後、焼鈍圧延を繰返し、第1図に示す如く、第1、7層のNi31wt%-Cuが0.004mm厚み、250mm幅、第3、5層のCuが0.009mm厚み、250mm幅、第2、6層のTiが0.02mm厚み、250mm幅、第4層のZrが0.034mm厚み、250mm幅からなる厚み0.1mmの複合箔ろう材を得た。

得られた7層の複合箔ろう材において、Zr材、Ti材、Cu材及びNi31wt%-Cu材の断面積比率は、

Zr材 34%、Ti材 40%、Cu材 18%、Ni31wt%-Cu材 8%であった。

複合箔ろう材より、それぞれ試験片を採取し、直径10mm、厚み5mm及び直径15mm、厚み5mm寸法の2枚のAl_2O_3セラミックス板間に介在させ、Ar雰囲気中にて900℃、10分の加熱を施して接合した後、第2図に示す如く、2枚のセラミックスを夫々反対方向に引張る条件にて剪断強度試験を行なった。その結果を第2表に示す。

また、比較のために、ろう材としてAgろうを用いて、セラミックス板にWをメタライズした後、実施例と同一寸法のAl_2O_3セラミックス板を接合して、H_2ガス雰囲気中にて850℃に5分間加熱した後、前記剪断強度試験を行なった。その結果を第2表に示す。

第2表

	ろう材	接合温度	剪断強度	メタライズ処理
本発明	Ni-Cu/Ti/Cu/Zr/Cu/Ti/Ni-Cu 複合箔ろう材	900℃	14.8 kgf/mm²	なし
比較例	銀ろう 85Ag-Cu合金	850℃	9.8 kgf/mm²	Wメタライズ

発明の効果

実施例より明らかな如く、従来例の如きメタライズ処理する必要がなく、加熱温度が900℃と比

較的低く、ろう付け作業性にすぐれ、高い接着強度が得られ、セラミックス同志のろう付けに最適の複合箔ろう材であることが分る。

また、複合箔であるため、被接着材の接合表面形状が複雑であっても容易にすぐれた接着ができる利点がある。

4.図面の簡単な説明

第1図はこの発明による複合箔ろう材の斜視説明図である。

第2図は剪断強度試験条件を示す被接着材の斜視説明図である。

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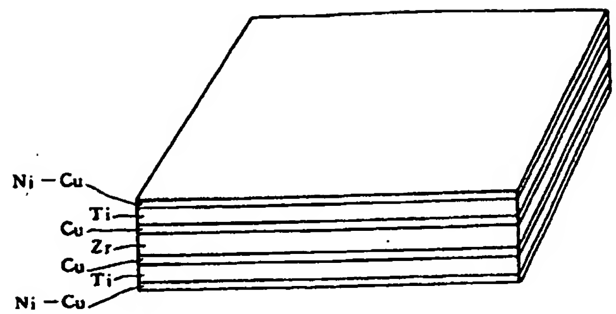
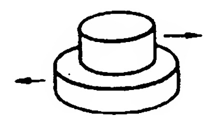
第1図



第2図



PTO 06-5317

Japanese Patent
H04-6173

COMPOSITE FOIL SOLDERING MATERIAL FOR JOINING CERAMICS
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UNITED STATES PATENT AND TRADEMARK OFFICE
Washington, D.C. June 2006

Translated by: Schreiber Translations, Inc.

Country	:	Japan
Document No.	:	H04-6173
Document Type	:	Kokai
Language	:	Japanese
Inventor	:	Kenji Hirano, Hiroshi Suenaga, and Masaaki Ishio
Applicant	:	Sumitomo Specialty Metal Co., Ltd.
IPC	:	C 04 B 37/00 B 23 K 35/14 B 32 B 15/01 C 22 C 9/06 19/03
Application Date	:	April 20, 1990
Publication Date	:	January 10, 1992
Foreign Language Title	:	Seramikku Setsugo Yo Fukugo Haku Ro Zai
English Title	:	COMPOSITE FOIL SOLDERING MATERIAL FOR JOINING CERAMICS

Specification

1. Title of the invention

COMPOSITE FOIL SOLDERING MATERIAL FOR JOINIGN CERAMICS

2. Patent Claim

1. A composite foil soldering material for joining ceramics characterized by the fact that it consists of a 7-layer composite material obtained by coating, on both planes of a Zr material provided as a core material, outer skin materials of a triple-layer laminate material comprising of Cu/Ti/Ni 20 ~ 70 wt%-Cu whereby the Ni 20 ~ 70 wt%-Cu material constitutes the outermost plane and that the respective cross-sectional area ratios of the aforementioned Zr material, Ti material, Cu material, & Ni 20 ~ 70 wt%-Cu material are confined to the following ranges:

Zr material: 30% ~ 40%;

Ti material: 35% ~ 45%;

Cu material: 10% ~ 20%;

Ni 20 ~ 70 wt%-Cu material: 5% ~ 15%.

3. Detailed explanation of the invention

(Industrial application fields)

The present invention concerns a composite foil soldering material for joining ceramics, and more specifically, a composite foil soldering material for joining ceramics capable, by virtue of the 7-layer composite foil soldering material constitution thereof obtained by coating, on both planes of a Zr material provided as a core material, outer skin materials of a triple-layer laminate material comprising of Cu/Ti/Ni 20 ~ 70 wt%-Cu, of ensuring not only an excellent soldering operative

[1] Numbers in the margin indicate pagination in the foreign text.

efficiency at a relatively low soldering operation temperature of 900°C but also a high adhesive strength.

(Prior art)

Generally speaking, in a case where ceramics are mutually joined for providing mechanical components, electric & electronic components, etc., their mutually opposing planes are metallized with metals bearing analogous thermal expansion coefficients & favorable wettabilities such as Mo, W, etc., and subsequently, the aforementioned metallized planes are mutually joined by means of Ag soldering, although immense labor & cost burdens are imposed on the metallizing & Ag soldering operations, and in particular, many problems are acknowledged in a case where heteromorphic ceramic sheets in possession of depressions & protrusions on the to-be-joined surfaces thereof are mutually joined.

The objective of the present invention, which has been conceived in acknowledgment of the foregoing current state of affairs, is to provide a soldering material which requires only a low soldering operation temperature on an occasion for mutually joining ceramics, which can be easily handled and therefore ensures an excellent adhesion operative efficiency, and which is capable not only of yielding a high adhesive strength but also of reducing the adhesion cost.

(Summary of the invention) /2

The present inventors conducted various investigations on foil-shaped soldering materials presumably easy to handle for purposes of reducing the cost of mutually joining ceramics provided as mechanical components, electric & electronic components, etc. and of providing a soldering material requiring only a low soldering operation temperature & bearing an excellent solderability, as a result of which the present invention has been completed after it had been discovered that a composite foil soldering material provided by specifiably combining a Ti material, a Zr material, a Cu material, & an Ni 20 ~ 70 wt%-Cu material and by designating specified cross-sectional area

ratios among them by means of press-down exhibits, as a soldering material for mutually joining ceramics provided as mechanical components, electric & electronic components, etc., an excellent solderability at a relatively high temperature and can be provided inexpensively.

In other words, the present invention concerns a composite foil soldering material for joining ceramics characterized by the fact that it consists of a 7-layer composite material obtained by coating, on both planes of a Zr material provided as a core material, outer skin materials of a triple-layer laminate material comprising of Cu/Ti/Ni 20 ~ 70 wt%-Cu whereby the Ni 20 ~ 70 wt%-Cu material constitutes the outermost plane and that the respective cross-sectional area ratios of the aforementioned Zr material, Ti material, Cu material, & Ni 20 ~ 70 wt%-Cu material are confined to the following ranges:

Zr material: 30% ~ 40%;

Ti material: 35% ~ 45%;

Cu material: 10% ~ 20%;

Ni 20 ~ 70 wt%-Cu material: 5% ~ 15%.

The composite foil soldering material of the present invention bears favorable wettabilities in relation to ceramics and is therefore ideal for mutually joining ceramics, and since preliminary ceramic metallizing processes indispensable for the likes of Ag soldering materials of the prior art are unnecessary and since the soldering operation temperature is relatively low at 900°C, its operative efficiency is excellent, and a high adhesive strength can be achieved by using this composite foil soldering material.

The composite foil soldering material of the present invention may, furthermore, be used for joining mutually homologous materials or mutually heterologous materials, and all conceivable oxide-type ceramics can be mutually joined.

(Constitution of the invention)

The composite foil soldering material of the present invention for joining ceramics is a 7-layer composite foil soldering material obtained by coating, on both planes of a Zr material provided as a core material, outer skin materials of a triple-layer laminate material comprising of Cu/Ti/Ni 20 ~ 70 wt%-Cu, and as Figure 1 indicates, the respective layers are, in a case where the uppermost plane layer & lowermost plane layer are designated respectively as first & seventh layers, laminated in the order shown in Table I below.

Table I

Triple-layer material (1st, 2nd, & 3rd layers)	Core material (4th layer)	Triple-layer material (5th, 6th, & 7th layers)
Ni-Cu-Ti-Cu	Zr	Cu-Ti-Ni-Cu

Zr is used as the core material of the fourth layer of the composite foil soldering material of the present invention not only because it contributes, as an active metal together with the Ti material within the triple-layer materials comprising the outer skin material (2nd & 6th layers), to the joining of ceramics but also because it is necessary for further lowering the melting point of Ti-Cu.

The Ti material (2nd & 6th layers) is, as has been mentioned above, necessary for achieving the requisite adhesive strength as a result of the reaction thereof, as an active metal, with ceramics.

Moreover, the Cu material is used as planes contacted with the Zr material provided as the core material of the fourth layer (3rd & 5th layers) in the present invention, for it is necessary for lowering the melting point of Ti.

Moreover, the Ni 20 ~ 70 wt%-Cu material is used as the outermost planes of the outer skin materials of the present invention (1st & 7th layers) because it is necessary not only for lowering the melting point of Ti but also for enhancing the joining strength of ceramics, and in a case where the

Ni ratio of the Ni-Cu material is lower than 20 wt%, an effect of improving the joining strength is absent, whereas in a case where said Ni ratio exceeds 70 wt%, it is undesirable in that the melting point of Ti cannot be lowered and that the operative temperature becomes elevated.

As far as the reasons why the cross-sectional area ratios of the Zr material (4th layer), Ti material (2nd & 6th layers), Cu material (3rd & 5th layers), & Ni 20 ~ 70 wt%-Cu material (1st & 7th layers) are limited are concerned, in a case where the Zr ratio is below 30%, where the Ti ratio is lower than 35%, where the Cu ratio exceeds 20%, and/or where the Ni 20 ~ 70 wt%-Cu ratio exceeds 15%, it is problematic in that it becomes difficult to induce a reaction with ceramics and that their joining becomes impossible, whereas in a case where the Zr ratio exceeds 40%, where the Ti ratio exceeds 45%, where the Cu ratio is lower than 10%, and/or where the Ni 20 ~ 70 wt%-Cu ratio is lower than 5%, it is undesirable in that the melting point becomes elevated and that the operative efficiency & ceramic wettability deteriorate. /9

The composite foil soldering material of the present invention may, for example, be obtained based on the following manufacturing method.

A Cu/Ti/Ni 20 ~ 70 wt%-Cu triple-layer clad sheet is prepared by uncoiling a coil-shaped Cu sheet & a coil-shaped Ni 20 ~ 70 wt%-Cu sheet and by flatly pressing, from respectively above & below, the aforementioned Cu sheet & Ni 20 ~ 70 wt%-Cu sheet onto a coil-shaped Ti sheet being uncoiled, as an intermediate layer. It is subsequently annealed.

Moreover, a 7-layer clad sheet is prepared by flatly pressing, from respectively above & below, Cu/Ti/Ni 20 ~ 70 wt%-Cu clad sheets onto the aforementioned coil-shaped Cu/Ti/Ni 20 ~ 70 wt%-Cu clad sheets, which is being uncoiled in a state where the coil-shaped Zr sheet, which serves as an intermediate layer, is also being uncoiled in such a way that the Ni 20 ~ 70 wt%-Cu material will prevail as the outermost plane.

Subsequently, it is repeatedly annealed & rolled, and a composite foil soldering material baring specified dimensions & specified cross-sectional area ratios thus becomes finished.

A foil with a thickness of 30 ~ 100 μm is appropriate as the composite foil soldering material of the present invention, for it is advantageous in the senses that it can easily fit various shapes of adhesion objects, that an excellent adhesive strength can accordingly be achieved, and that the adhesion operation also becomes facilitated.

It becomes possible, furthermore, to significantly improve the ceramic wettability of the composite foil soldering material of the present invention in a case where it is coated with an Ag material.

(Application examples)

A Ti sheet with a thickness of 0.4 mm & a width of 250 mm, a Cu sheet with a thickness of 0.18 mm & a width of 250 mm, and a Ni 31 wt%-Cu sheet with a thickness of 0.08 mm & a width of 250 mm were, in a state where they were being uncoiled and where the Cu sheet & Ni 31 wt%-Cu sheet were being contacted with both planes of the Ti sheet provided as a core, rolled at a rolling ratio of 65%, as a result of which a Cu/Ti/Ni 31 wt%-Cu clad sheet with a thickness of 0.23 mm & a width of 250 mm was obtained. It was subsequently subjected to diffusive annealing for the purpose of upping the joining strength of the press contact interface.

Next, a 7-layer clad sheet with a thickness of 0.25 mm & a width of 250 mm was obtained by rolling, at a rolling ratio of 65%, a Zr sheet with a thickness of 0.24 mm & a width of 250 mm & a pair of the aforementioned triple-layer Cu/Ti/Ni 31 wt%-Cu clad sheet with a thickness of 0.23 mm & a width of 250 mm in a state where said triple-layer clad sheets were being contacted with both planes of the Zr sheet provided as a core in such a way that the Ni 31 wt%-Cu material will prevail as the outermost plane.

It was subsequently annealed & rolled repeatedly, as a result of which a composite foil soldering material with a thickness of 0.1 mm comprising, as each of first & seventh layers, of an Ni 31 wt%-Cu layer with a thickness of 0.004 mm & a width of 250 mm, as each of third & fifth layers, of a Cu layer with a thickness of 0.009 mm & a width of 250 mm, as each of second & sixth

layers, a Ti layer with a thickness of 0.02 mm & a width of 250 mm, and, as the fourth layer, a Zr layer with a thickness of 0.034 mm & a width of 250 mm was obtained, as Figure 1 indicates.

The respective cross-sectional area ratios of the Zr material, Ti material, Cu material, & Ni 31 wt%-Cu material within the 7-layer composite foil soldering material thus obtained were as follows:

Zr material: 34%; Ti material: 40%; Cu material: 18%; Ni 31 wt%-Cu material: 8%.

After a testpiece had been sampled from the composite foil soldering material, it was sandwiched in-between a pair of Al_2O_3 ceramic sheets with a diameter of 10 mm & a thickness of 5 mm and a diameter of 15 mm & a thickness of 5 mm, and after the obtained structure had been heated & joined within an Ar atmosphere at 900°C over a 10-min. period, it was subjected to a shear strength test under conditions where the pair of ceramic sheets were pulled apart into mutually opposing directions according to the illustration of Figure 2. The results are shown in Table II.

For comparative purposes, furthermore, Al_2O_3 ceramic sheets dimensionally identical to their counterparts of the application example were metallized with W and then mutually joined with an Al solder provided as a soldering material, and after the joined structure had subsequently been heated within an H_2 atomosphere at 850°C over a 5-min. period, it was subjected to the aforementioned shear strength test. The results are shown in Table II.

Table II

	Soldering material	Joining temperature	Shear strength	Metallizing treatment

Present invention	Ni-Cu/ Ti/Cu/Zr/Cu/ Ti/Ni-Cu composite foil soldering material	900°C	14.8 kgf/mm^2	None
Comparative Example	Silver solder (85Ag-Cu alloy)	850°C	9.8 kgf/mm^2	W metallization

(Effects of the invention)

As the application example clearly indicates, the present composite foil soldering material can be acknowledged to be ideal for mutually soldering ceramics in that it requires no metallizing treatment indispensable in the prior art, that the heating temperature is relatively low at 900°C, that it ensures an excellent soldering operative efficiency, and that a high adhesive strength can be /4 achieved.

The present composite foil form, furthermore, is advantageous in that an excellent adhesion state can easily be achieved even in a case where the joining surface shapes of adhesion targets are complex.

4. <u>Brief explanation of the figures</u>

Figure 1 is an oblique view demonstrational diagram pertaining to the composite foil soldering material of the present invention.

Figure 2 is an oblique view demonstrational diagram pertaining to adhesion targets provided for showing shear strength test conditions.

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Figure 1

第1図

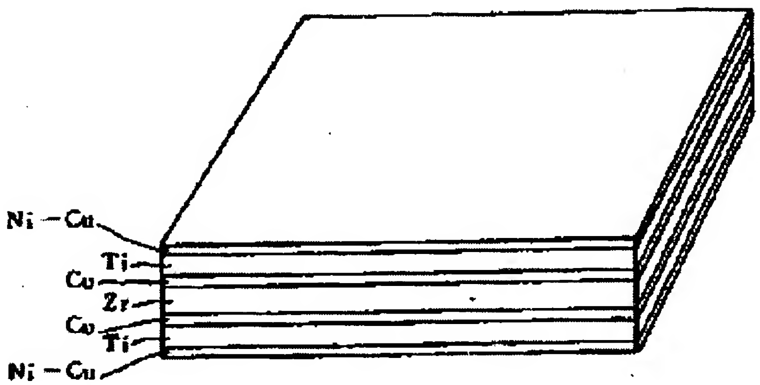

Figure 2

第2図